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Jawaharlal Nehru

“Step Out From the Old to the New”

IS 4566 (1979): Methylene Chloride (dichloromethane),
Technical [PCD 9: Organic Chemicals Alcohols and Allied
Products and Dye Intermediates]



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Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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REAFFIRMED

IS : 4566 - 1979

2006

Indian Standard

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SPECIFICATION FOR METHYLENE
CHLORIDE (DICHLOROMETHANE),
TECHNICAL

(*First Revision*)

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

September 1980

Indian Standard
**SPECIFICATION FOR METHYLENE
 CHLORIDE (DICHLOROMETHANE),
 TECHNICAL
 (First Revision)**

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(Continued on page 2)

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AMENDMENT NO. 1 JANUARY 1998
TO
IS 4566 : 1979 SPECIFICATION FOR METHYLENE
CHLORIDE (DICHLOROMETHANE), TECHNICAL
(First Revision)

[Page 4, clause 2.1(b)] — Substitute 'and other applications' for 'purposes'.

[Page 5, Table 1, *Sl No.* (iii), col 2] — Substitute 'Residue on evaporation' for 'Non-volatile matter'.

[Page 5, Table 1, *Sl No.* (v), col 4] — Substitute '0.0005' for '0.01'.

[Page 14, clause A-3.3.2(a) and (b)] — Substitute the following for the existing matter:

- a) For every 10 mm above 760 mmHg pressure, subtract 0.38°C from the observed temperature of the boiling range to get the specified temperature as specified in Table 1.
- b) For every 10 mm above 760 mmHg pressure, add 0.38°C from the observed temperature of the boiling range to get the specified temperature range as specified in Table 1.

(Page 14, clause A-4.2) — Substitute the following for the existing clause:

'A-4.2 Procedure

Clean the basin and heat in the oven at 105°C to 110°C for 30 minutes and dry in desiccator. Weigh it to the nearest 0.000 2 g (*M*₁) after 30 minutes. Measure out 100 ml of the material in the basin (*V*). Evaporate the material over a water-bath in a fume cupboard to practically dryness. Further heat in the oven at 105°C to 110°C for 30 minutes. Cool in a desiccator for 30 minutes and weigh accurately. Repeat the heating for 15 minutes followed by cooling and weighing till constant weight is attained.'

(Page 15, clause A-4.3) — Substitute the following for the existing clause:

A-4.3 Calculation

$$\begin{array}{l} \text{Residue on evaporation,} \\ \text{percent, by mass} \end{array} = \frac{M}{d}$$

Amend No. 1 to IS 4566 : 1979

where

M = mass in g of the residue obtained, and

d = relative density of the material.

(*Page 15, clause A-5.1*) — Substitute '50' for '20'.

(*Page 15, clause A-6.1.1*) — Substitute the following for the existing clause:

A-6.1.1 Bromothymol Blue Indicator

Dissolve 0.5 g of water soluble bromothymol blue crystal in 100 ml of water. Dilute 80 ml of this solution to 500 ml with methyl alcohol.

(*Page 15, clause A-6.2*) — Substitute the following for the existing clause:

'A-6.2 Procedure

To 100 ml of water, neutral to bromothymol blue, add 100 ml of the material and shake vigorously for one minute at least. Add bromothymol blue indicator and titrate with standard sodium hydroxide solution until a bluish green end point persists for 1 minute.'

(*Page 17, clause A-9.2.3*) — Insert 'wt/vol' at the end of the sentence.

[*Page 20, clause A-14.3.1(g)*] — Insert 'microlitre' after sample size.

(*Page 21, Appendix B*) — Delete 'PURE AND ' from the caption.

(PCD 9)

**AMENDMENT NO. 2 MAY 2002
TO
IS 4566 : 1979 SPECIFICATION FOR METHYLENE
CHLORIDE (DICHLOROMETHANE), TECHNICAL
(*First Revision*)**

[*Page 5, Table 1, Sl No. (v), col 4*] — Substitute '0.001' for '0.01'

(PCD 9)

Reprography Unit, BIS, New Delhi, India

Indian Standard
SPECIFICATION FOR METHYLENE
CHLORIDE (DICHLOROMETHANE),
TECHNICAL
(*First Revision*)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 30 October 1979, after the draft finalized by the Organic Chemicals (Miscellaneous) Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 This standard was first published in 1968. With a view to making it appropriate to meet the current needs of the industry, the Committee made a detailed review of the standard and decided to revise it. In the present revision the requirements for acidity have been modified for the industrial grade of the material. Also another grade suitable for the manufacture of refrigerants has been added and the methods of tests modified suitably.

0.3 Methylene chloride (dichloromethane*) finds extensive use as a major component of non-flammable paint removers; in the manufacture of photographic films; solvent for alkaloids, bitumens, crude rubber, oils, resins, waxes and many organic compounds; as solvent mixtures for cellulose esters and ethers, textile and leather coatings, lacquers, fire-extinguishing compositions; refrigeration; local anaesthetic in dentistry; as spotting agent; degreasing and dewaxing agent; in chemical syntheses; as a vapour pressure depressant in aerosols; and various other uses in the chemical industry.

0.4 Though least toxic of the chloromethanes, methylene chloride is strongly narcotic causing dizziness and slight nausea. It is very dangerous to the eyes. In view of this and its great volatility, care should be taken in its use. The place where methylene chloride is handled and stored should be well ventilated and it should not be brought in contact with hot surfaces and naked flame (*see also 4.1.1, 4.2.1 and 4.2.2*).

0.5 In the preparation of this standard due weightage has been given to international co-ordination among the standards and practices prevailing in different countries in addition to relating it to the practices in the country.

*International name is dichloromethane as decided by the International Union of Pure and Applied Chemistry (IUPAC).

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This has been met by deriving assistance from BS 1994 : 1953 'Dichloromethane (methylene chloride)' published by the British Standards Institution and ISO 1869-1977 Methylene chloride 'List of methods of test' issued by the International Organization for Standardization (ISO).

0.6 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for methylene chloride (dichloromethane), technical.

1.1.1 This standard does not apply to the material intended for pharmaceutical use.

2. GRADES

2.1 The material shall have two grades, namely:

- a) Grade 1 — for the use in photofilm industry, and
- b) Grade 2 — suitable for industrial purposes.

3. REQUIREMENTS

3.1 Description — The material shall be a colourless liquid free from matter in suspension and shall have a sweet penetrating ether-like odour. It shall be soluble in ethyl alcohol and ethyl ether in all proportions.

3.2 The material shall also comply with the requirements prescribed in Table 1, when tested according to the methods given in Appendix A. Reference to the relevant clauses of Appendix A is given in col 5 of the table.

4. PACKING AND MARKING

4.1 Packing

4.1.1 Subject to the provisions of the Red Tariff No. 18 of 1960 issued by the Indian Railways Conference Association with any additions and alterations made thereafter, the material shall be packed in fully air-tight well-closed galvanized iron containers capable of withstanding a pressure of 0.5

*Rules for rounding off numerical values (*revised*).

bar depending upon the maximum temperature to which it would be exposed. The containers shall have preferably replaceable closure. Any other suitable container compatible with the properties of the material may also be used.

**TABLE 1 REQUIREMENTS FOR METHYLENE CHLORIDE
(DICHLOROMETHANE), PURE AND TECHNICAL**
(Clause 3.2)

Sl. No.	CHARACTERISTIC	REQUIREMENT		METHOD OF TEST (REF TO CLAUSE No. IN APPENDIX A)
		Grade 1	Grade 2	
(1)	(2)	(3)	(4)	(5)
i)	Relative density* at 27/27°C	1.314 to 1.319†	1.310 to 1.320	A-2
ii)	Distillation range at a pressure of 1 bar	2 to 97 percent by volume shall distil between 39 to 40.5°C	2 to 97 percent by volume shall distil between 39 to 41°C	A-3
iii)	Non-volatile matter, percent by mass, <i>Max</i>	0.001	0.01	A-4
iv)	Moisture, percent by mass, <i>Max</i>	0.02	0.05	A-5
v)	Acidity (as HCl), percent by mass, <i>Max</i>	0.000 3	0.01	A-6
vi)	Free chlorine	To pass test	To pass test	A-7
vii)	Aldehydes	To pass test	—	A-8
viii)	Iron (as Fe), parts per million, <i>Max</i>	0.04	—	A-9
ix)	Heavy metals	To pass test	—	A-10
x)	Resistance to potassium permanganate	To pass test	—	A-11
xi)	Nessler's reagent	To pass test	—	A-12
xii)	Resistance to sulphuric acid	To pass test	—	A-13
xiii)	Total impurities due to methyl chloride and chloroform, percent by mass, <i>Max</i>	0.1	—	A-14

*Relative density is the term adopted for specific gravity with water as reference substance by the International Organization for Standardization (ISO). This term is synonymous with specific gravity.

†The correction factor within the range 15 to 35° is +0.001 6 for each degree Celsius fall and -0.001 6 for each degree Celsius rise in temperature.

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4.2 Marking

4.2.1 The containers shall be suitably marked in red letters as POISON-
OUS and shall be labelled as shown in Fig. 5 of IS : 1260 (Part I)-1973*.
The label shall also bear the following clear and legible instruction:

‘STORE IN A COOL, WELL VENTILATED PLACE AWAY
FROM FIRE AND FLAMES. USE WITH ADEQUATE
VENTILATION. AVOID PROLONGED BREATHING OF
VAPOURS’

4.2.2 The containers shall be marked with the name of the material; its
net mass; name of the manufacturer and recognized trade-mark, if any;

4.2.3 The containers may also be marked with the ISI Certification
Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the
Indian Standards Institution (Certification Marks) Act and the Rules and Regulations
made thereunder. The ISI Mark on products covered by an Indian Standard conveys
the assurance that they have been produced to comply with the requirements of that
standard under a well-defined system of inspection, testing and quality control which is
devised and supervised by ISI and operated by the producer. ISI marked products are
also continuously checked by ISI for conformity to that standard as a further safeguard.
Details of conditions under which a licence for the use of the ISI Certification Mark may
be granted to manufacturers or processors, may be obtained from the Indian Standards
Institution.

5. SAMPLING

5.1 Representative samples of the material shall be drawn and their con-
formity to this standard shall be judged as prescribed in Appendix B.

APPENDIX A

(Clause 3.2, and Table 1)

METHODS OF TEST FOR METHYLENE CHLORIDE, PURE AND TECHNICAL

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (see
IS : 1070-1977†) shall be used in tests.

NOTE — ‘ Pure chemicals ’ shall mean chemicals that do not contain impurities which
affect the results of analysis.

*Pictorial markings for handling and labelling of goods: Part I Dangerous goods (*first
revision*).

†Specification for water for general laboratory use (*second revision*).

A-2. DETERMINATION OF RELATIVE DENSITY

A-2.0 General — Relative density is determined using specific gravity bottle or pyknometer.

A-2.1 Apparatus

A-2.1.1 Specific Gravity Bottle or Pyknometer — with well-fitting ground-glass joints. To calibrate, clean and dry the bottle or pyknometer thoroughly, weigh and then fill with water recently boiled and cooled to below 27°C after removing the cap of the side arm. Fill to overflowing by holding the bottle or pyknometer on its side in such a manner as to prevent entrapment of air bubbles, insert the stopper and immerse in a water bath at $27 \pm 0.2^\circ\text{C}$. Keep the entire bulb completely covered with water and hold at that temperature for 30 minutes. Carefully remove any water which has exuded from the capillary opening. Remove from the bath, wipe completely dry, replace the cap, cool to a lower temperature and weigh. Calculate the mass of water. The constant gravity bottle of about 50 ml capacity and of either of the two shapes as shown in Fig. 1 is recommended.

A-2.1.2 Water Bath—maintained at $27 \pm 0.2^\circ\text{C}$.

A-2.1.3 Thermometer — any convenient thermometer of a suitable range with 0.1 or 0.2°C subdivisions. The thermometer should be checked against a standard thermometer, which has been calibrated and certified by the National Physical Laboratory, New Delhi, or any other organization recognized for such work (*see also* IS : 4825-1968*).

A-2.2 Procedure — Fill the bottle or pyknometer with the material previously kept at about 27°C to overflowing, holding the apparatus on its side in such a manner as to prevent entrapment of air bubbles after removing the cap of the side arm. Insert the stopper, immerse in the water bath at $27 \pm 0.2^\circ\text{C}$, and hold for 30 minutes. Carefully wipe off any liquid which has come through the capillary opening. Remove the apparatus from the bath, clean and dry it thoroughly. Replace the cap, cool to a lower temperature and weigh.

A-2.3 Calculation

$$\text{Relative density at } 27/27^\circ\text{C} = \frac{A - B}{C - B}$$

where

A = mass of the specific gravity bottle or pyknometer filled with the material at 27°C,

B = mass of the specific gravity bottle or pyknometer, and

C = mass of the specific gravity bottle or pyknometer filled with water at 27°C.

*Specification for laboratory and reference thermometers.

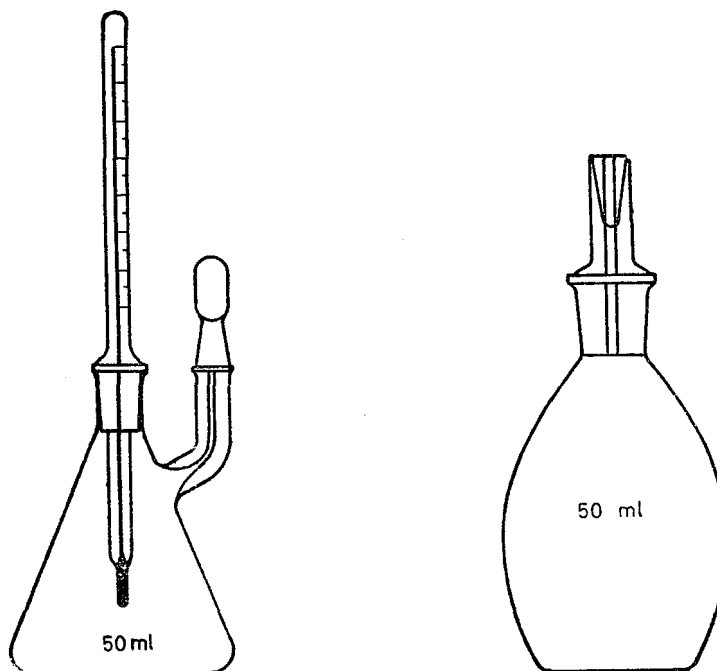


FIG. 1 SPECIFIC GRAVITY BOTTLES

A-3. DETERMINATION OF DISTILLATION RANGE

A-3.0 General—The fraction distilling at specified temperature range is collected and measured.

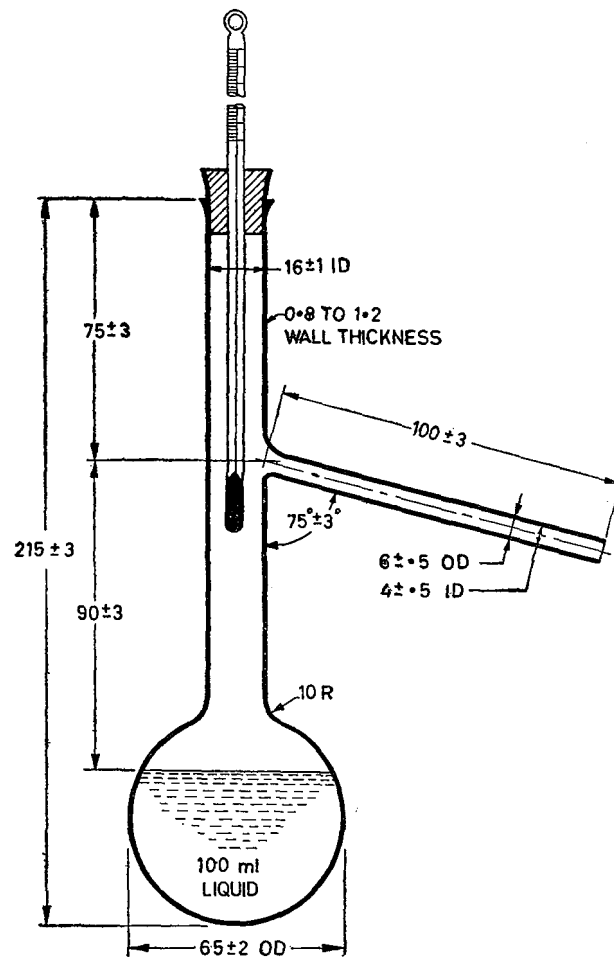
A-3.1 Apparatus

A-3.1.1 Distillation Flask— of the shape and dimensions shown in Fig. 2. Fix the flask in the vertical position by means of a clamp at the extreme upper end of the neck.

A-3.1.2 Thermometer— A suitable partial immersion thermometer having a range of 39 to 51°C, graduated at each 0.2°C, with a maximum scale error of 0.2 deg C (see also IS : 4825-1968*).

A-3.1.2.1 Position of thermometer— The thermometer shall be held concentrically in the neck of the flask by means of a well-fitting stopper of a material which is not attacked by the liquid, and the junction of the

*Specification for laboratory and reference thermometers.



All dimensions in millimetres.

FIG. 2 DISTILLATION FLASK

capillary tube and the main bulb of the thermometer shall be maintained level with the lower edge of the joint of the side-tube and the neck of the flask. The stopper shall project about 10 mm above the top of the neck of the flask. When the thermometer is fixed in position as indicated above, the immersion line on the thermometer shall be in the neighbourhood of the top of the cork holding the thermometer.

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A-3.1.2.2 The thermometer shall bear a certificate of the National Physical Laboratory, New Delhi, or any other organization recognized for such work.

A-3.1.3 Rectangular Draught Screen — made of 0.8 mm thick sheet metal, with the dimensions shown in Fig. 3 and open at the top and bottom, fitted with hard asbestos board, with a central hole of 100 mm diameter. It shall comply with the following requirements:

- a) In each of the two narrow sides of the draught screen there shall be two circular holes, each 25 mm in diameter, and in each of the four sides of the draught screen there shall be three holes with their centres 25 mm above the base of the draught screen. These holes shall occupy the position shown in Fig. 3. The diameter of each of the holes centrally situated in the longer sides shall be 25 mm and of the remaining ten holes shall be 12.5 mm. At the middle of each of the wider sides a vertical slot with the dimensions shown in Fig. 3 shall be cut downwards from the top of the screen.
- b) A sheet of hard asbestos, 6 mm in thickness, and having a central circular hole 50 mm in diameter, shall be supported horizontally in the screen and shall fit closely to the sides of the screen to ensure that hot gases from the source of heat do not come in contact with the sides or neck of the flask. The supports for this asbestos sheet may conveniently consist of triangular pieces of metal sheet firmly fixed to the screen at its four corners.
- c) In one of the narrow sides of the screen a door shall be provided having the dimensions and position as shown in Fig. 3. In each of the narrow sides of the screen a mica window shall be placed centrally, with the bottom of the window on a level with the top of the asbestos shelf. The dimensions and position of the windows are shown in Fig. 3.

A-3.1.4 Liebig Condenser — with the bent end made of Type 1 glass conforming to IS : 2303-1963*, with a wall thickness of 1.0 to 1.5 mm and conforming to the shape and dimensions shown in Fig. 4.

A-3.1.4.1 Alternatively, the bent portion may be substituted by an adapter fitted externally in such a manner that distillate does not come in contact with the cork.

A-3.1.5 Receiver or Graduated Cylinder — 100 ml capacity, with millilitre marks running a half way round the circumference, and 5-millilitre marks running three-quarters-way round and 10-millilitre marks running all round the circumference, and numbered as shown in Fig. 5. Any other graduated 100-ml cylinder may also be used.

*Method of grading glass for alkalinity.

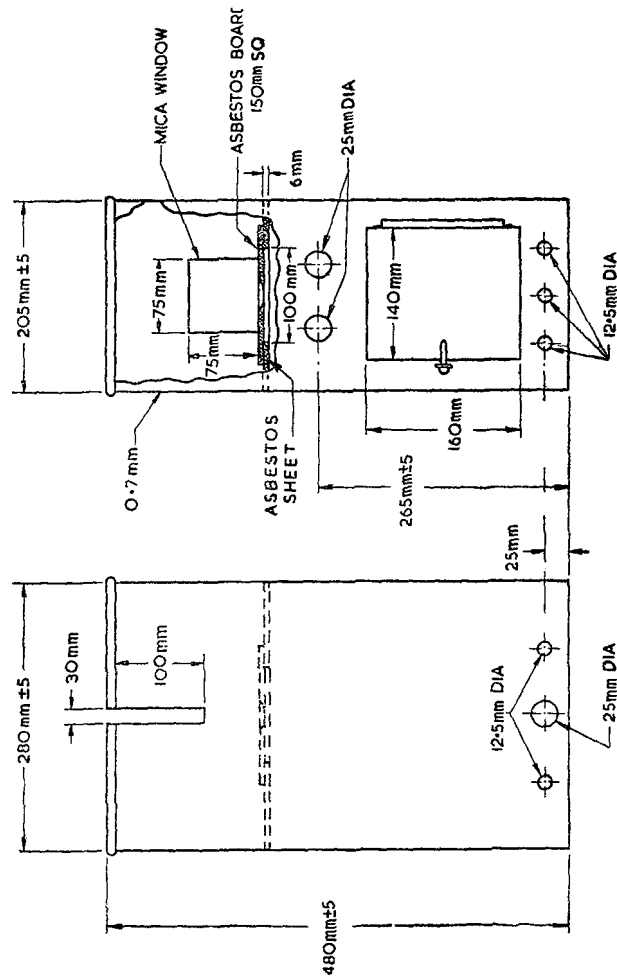


FIG. 3 RECTANGULAR DRAUGHT SCREEN

IS: 4566 - 1979

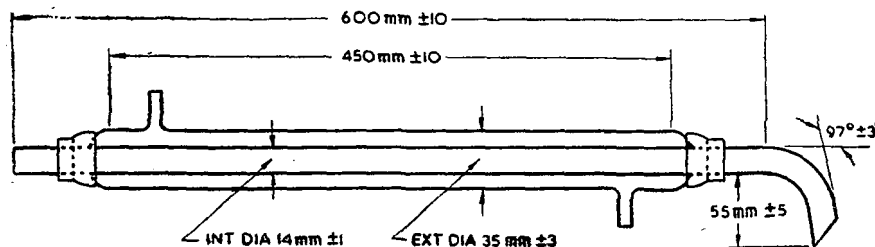


FIG. 4 LIEBIG CONDENSER

A-3.1.6 Water Bath — thermostatically controlled.

A-3.1.7 Half-Second Pendulum — for measuring the rate of distillation.

A-3.2 Procedure — Assemble the apparatus as shown in Fig. 6. Measure 100-ml of the material at laboratory temperature by means of 100-ml receiver or graduated cylinder and transfer it to the distillation flask. Add a fragment (about 2 mm cube) of porous or other suitable inert material to prevent bumping. Connect the flask to the condenser and insert the thermometer. Pass an adequate supply of cooling water at 0 to 5°C through the condenser. To receive the distillate, use the receiver in which the sample was measured, without rinsing or drying. Heat the flask slowly especially after ebullition has begun, in order that the mercury column of the thermometer may become fully expanded before the first drop of distillate falls into the receiver, care being taken that the total period of this preliminary heating shall be for not less than 5 nor greater than 10 minutes. Place the receiver so that the condensate will flow down its side. Continue the distillation at the rate of 4 to 5 ml per minute (about 2 drops per second). Read the volume of distillate in the receiver when the thermometer indicates each of the specified distillation temperatures, the temperatures on the thermometer scale being corrected as specified under A-3.3. The difference between the volumes so recorded is the percentage by volume distilling between the specified temperatures at 760 mm pressure.

A-3.3 Correction of the Thermometer Reading — The following correction shall be applied before starting distillation.

A-3.3.1 Error of Scale — In all the thermometer readings, make the corrections as indicated on the certificate of the instrument.

A-3.3.2 Correction for Barometric Pressure — If the barometric pressure prevailing during the determination is normal, namely, 760 mmHg, no correction need be applied to the specified temperature and the thermometer scale as corrected under A-3.3.1 shall be used as such. If, however, the

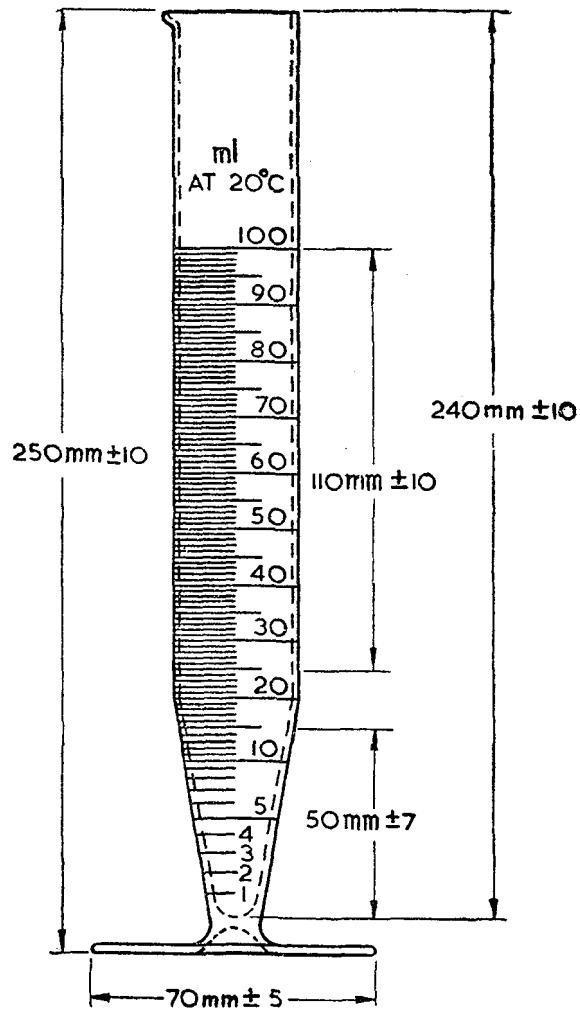


FIG. 5 RECEIVER OR GRADUATED CYLINDER

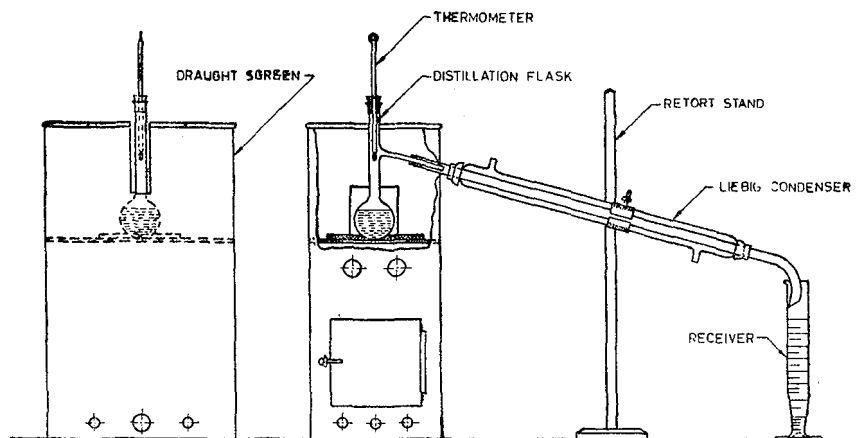


FIG. 6 ASSEMBLY OF APPARATUS FOR DETERMINING DISTILLATION RANGE

prevailing pressure deviates from 760 mmHg, the specified temperature shall also be corrected as follows:

- a) For every 10 mm above 760 mmHg, subtract 0.38°C from the specified temperature; and
- b) For every 10 mm below 760 mmHg, add 0.38°C to the specified temperature.

A-4. DETERMINATION OF RESIDUE ON EVAPORATION

A-4.1 Apparatus

A-4.1.1 *Porcelain Basin* — 150 ml capacity [see IS : 2837 (Part II)-1977*]

A-4.1.2 *Water-Bath*

A-4.1.3 *Oven* — capable of maintaining temperature between 105°C to 110°C .

A-4.2 *Procedure* — Clean the basin and dry in a desiccator. Weigh it to the nearest 0.0002 g . Take 100 ml of the material in the basin and weigh again to the nearest 0.01 g . Evaporate the material over a water-bath in a fume cupboard to practically dryness. Further heat in the oven at 105 to 110°C for 30 minutes. Cool in a desiccator and weigh accurately. Repeat the heating for 15 minutes, followed by cooling and weighing till two consecutive weighings do not differ by more than 0.5 mg .

*Specification for porcelain crucibles and basins: Part II Basins (*first revision*).

A-4.3 Calculation

$$\begin{array}{l} \text{Residue on evaporation, percent} \\ \text{by mass} \end{array} = \frac{M_2 - M}{M_1 - M} \times 100$$

where

M_2 = mass in g of the basin with the dry residue,

M = mass in g of the basin, and

M_1 = mass in g of the basin with 100 ml of the material.

A-5. DETERMINATION OF MOISTURE CONTENT

A-5.0 General — Moisture content is determined using the Karl Fischer method.

A-5.1 Take 20 ml of the material and determine the moisture content as described in IS : 2362-1973*.

A-6. DETERMINATION OF ACIDITY

A-6.0 General — Acidity is determined by titrating a known quantity of material with a standard alkali.

A-6.1 Reagents

A-6.1.1 Phenolphthalein Indicator Solution — Prepare the indicator by dissolving 0.5 g of the phenolphthalein in 100 ml of ethanol (95 percent) and make faintly pink by the addition of dilute sodium hydroxide solution.

A-6.1.2 Standard Sodium Hydroxide Solution — 0.01 N.

A-6.2 Procedure — Place 100 ml of freshly boiled and cooled distilled water (neutralized to phenolphthalein indicator solution) in a 300-ml glass-stoppered conical flask. Add 100 ml of the methylene chloride and shake vigorously. Allow the layers to separate, add 0.5 ml of the phenolphthalein indicator and titrate with 0.01 N sodium hydroxide using a micro-burette to the end point.

A-6.3 Calculation

$$\begin{array}{l} \text{Acidity (as HCl), percent} \\ \text{by mass} \end{array} = 3.65 \frac{V_1 N}{V D}$$

*Method for determination of water by the Karl Fischer method (*first revision*).

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where

V_1 = volume in millilitres of standard sodium hydroxide solution,

N = normality of standard sodium hydroxide solution,

V = volume in millilitres of the material taken for the test, and

D = relative density of the material.

A-7. TEST FOR FREE CHLORINE

A-7.0 Outline of the Method — The material is shaken with a 3,3'-dimethylnaphthidine solution and the colour developed, if any, is noted.

A-7.1 Apparatus

A-7.1.1 Graduated Measuring Cylinder — 50 ml, glass stoppered (see IS : 878-1975*).

A-7.2 Reagent

A-7.2.1 3,3'-Dimethylnaphthidine Solution — Dissolve 0.01 g of finely ground 3,3'-dimethylnaphthidine in 5 ml of glacial acetic acid and dilute rapidly with water to 200 ml. Store the solution in the dark.

A-7.3 Procedure — To 50 ml of the material contained in the graduated measuring cylinder, add 5 ml of the 3,3'-dimethylnaphthidine solution and shake the cylinder for 30 seconds.

A-7.3.1 The material shall be considered as showing no free chlorine if no pink colour is developed.

A-8. DETERMINATION OF ALDEHYDES

A-8.0 General — The material is mixed with water and the aqueous layer is tested with silver ammonium nitrate solution to determine the presence of aldehydes.

A-8.1 Reagents

A-8.1.1 Silver Nitrate Solution — Dissolve 1 g of silver nitrate in 20 ml of water.

A-8.1.2 Ammonium Hydroxide Solution — 10 percent (v/v).

A-8.1.3 Silver Ammonium Nitrate Solution — Add ammonium hydroxide solution drop by drop to silver nitrate solution until the precipitate first

*Specification for graduated measuring cylinders (first revision).

formed is dissolved completely leaving a clear solution. Do not add excess ammonium hydroxide.

A-8.2 Procedure — Shake 50 ml of the sample with 50 ml of distilled water in a separating funnel for 5 minutes. Allow the layers to separate and draw off the methylene chloride layer and discard. Collect the aqueous layer in a measuring jar. Take 10 ml of the aqueous layer in a test tube, add 5 ml of silver ammonium nitrate solution and shake well. Keep the test tube in dark for 15 minutes.

A-8.2.1 The material shall be taken to have passed the test if no brown colour or turbidity is developed.

A-9. DETERMINATION OF IRON

A-9.0 General — The material is evaporated and the residue is treated with ammonium thiocyanate solution.

A-9.1 Apparatus

A-9.1.1 Nessler Cylinders — 100 ml capacity.

A-9.2 Reagents

A-9.2.1 Concentrated Hydrochloric Acid — (see IS : 265-1976*).

A-9.2.2 Ammonium Persulphate

A-9.2.3 Ammonium Thiocyanate Solution — approximately 10 percent.

A-9.2.4 Concentrated Sulphuric Acid — (see IS : 266-1977†).

A-9.2.5 Standard Iron Solution — Dissolve 0.702 g of ferrous ammonium sulphate [$\text{FeSO}_4 (\text{NH}_4)_2 \text{SO}_4 \cdot 6\text{H}_2\text{O}$] in 100 ml of distilled water and 10 ml of concentrated sulphuric acid and dilute with water to 1 000 ml mark. Transfer 100 ml of this solution to 1 000-ml volumetric flask and dilute again to 1 000 ml. One millilitre of this solution is equivalent to 0.01 mg of iron (as Fe). Further dilute 100 ml of this solution to 1 000 ml. One ml of this diluted solution is equivalent to 0.001 mg of iron (as Fe).

A-9.3 Procedure — Dissolve the residue from A-4.2 with one ml concentrated hydrochloric acid and 20 ml water by warming and then make up to 100 ml with water. Take 50 ml of this solution into a Nessler cylinder, add 30 mg of ammonium persulphate and one ml of ammonium thiocyanate solution. Carry out a control test in another Nessler cylinder using the same reagents in the same total volume using 2.5 ml of standard iron solution in place of the material.

*Specification for hydrochloric acid (second revision).

†Specification for sulphuric acid (second revision).

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A-9.3.1 The material shall be taken to have passed the test if the intensity of the colour produced with the material is not greater than that produced in the control test.

A-10. DETERMINATION OF HEAVY METALS

A-10.0 General — Heavy metals are detected by treating with hydrogen sulphide water.

A-10.1 Apparatus

A-10.1.1 Nessler Cylinder — 100 ml capacity.

A-10.2 Reagent

A-10.2.1 Hydrogen Sulphide Water — Pass hydrogen sulphide gas through water and prepare a saturated solution of hydrogen sulphide water.

A-10.3 Procedure — To another 25 ml sample of aqueous extract from A-9.3, taken in a Nessler cylinder, add 25 ml of water and 5 ml of hydrogen sulphide water.

A-10.3.1 The material shall be taken to have passed the test if the colour formed is not greater than the blank similarly prepared.

A-11. TEST FOR RESISTANCE TO POTASSIUM PERMANGANATE

A-11.0 General — The material is treated with potassium permanganate and examined for its resistance.

A-11.1 Reagent

A-11.1.1 Potassium Permanganate Solution — 0.1 percent solution.

A-11.2 Procedure — Take 20 ml of the sample into a stoppered measuring cylinder. To this solution add 0.2 ml of potassium permanganate solution. Mix well and keep aside the cylinder in an ice-bath for 30 minutes.

A-11.2.1 The material shall be taken to pass the test if the pink colour does not entirely disappear.

A-12. NESSLER'S REAGENT TEST

A-12.0 General — The aqueous extract of the sample is treated with Nessler's reagent and tested for the presence of ammonia and aldehydes.

A-12.1 Reagent**A-12.1.1 Nessler's Reagent**

A-12.2 Procedure — To another 20 ml of aqueous extract from A-8.2 taken in a stoppered cylinder, add 5 ml of Nessler's reagent. Shake well and leave for 5 minutes.

A-12.2.1 The material shall be taken to have passed the test if no turbidity in yellow, red or black colour is formed.

A-13. TEST FOR RESISTANCE TO SULPHURIC ACID

A-13.0 General — The material is treated with sulphuric acid and tested for the presence of carbonizable impurities.

A-13.1 Reagent

A-13.1.1 Concentrated Sulphuric Acid — (see IS : 266-1977*).

A-13.2 Procedure — To 20 ml of the sample in a stoppered measuring cylinder, carefully add 5 ml of concentrated sulphuric acid. Mix well and keep aside for 5 minutes.

A-13.2.1 The material shall be taken to have passed the test if the colour of the acid layer is not more than faint yellow.

A-14. DETERMINATION OF TOTAL IMPURITIES DUE TO METHYL CHLORIDE AND CHLOROFORM

A-14.0 General — The presence and estimation of methyl chloride and chloroform in the sample is determined by chromatographic analysis.

A-14.1 Apparatus

A-14.1.1 Gas Chromatograph — Any commercially available gas chromatograph with a thermal conductivity detector may be used. The operating conditions of one of the suitable columns are given below:

Column

Material	Stainless steel
Length	3.6 m
Internal diameter	4 mm, approximately
External diameter	6 mm, approximately
Support	Chromosorb W (250-micron IS Sieve)
Stationary phase	20 percent silicone GESF — 96
Preparation of the stationary phase and support	20 percent by mass of silicone (GESF 96) on Chromosorb in (250-micron IS Sieve)

*Specification for sulphuric acid (second revision).

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A-14.1.2 Microlitre Syringe — With fixed needle 5 cm long, 50 (microlitre) capacity.

A-14.1.3 Recorder — Minus 0.05 to plus 1.05 mV; 25 cm strip chart; response 1 second full scale; and chart speed of 12.5 mm/min.

A-14.2 Reagent

A-14.2.1 Acetone

A-14.2.2 Synthetic Standard of Pure Methylene Chloride — Containing known amounts of methyl chloride and chloroform.

A-14.3 Procedure

A-14.3.1 Helium or hydrogen may be used as the carrier gas. The operating parameters of the gas chromatograph when using helium or hydrogen shall be as follows:

	<i>Helium</i>	<i>Hydrogen</i>
a) Detector block temperature, °C	265°C	265°C
b) Injection port temperature, °C	185°C	185°C
c) Column oven temperature, °C	80°C	80°C
d) Bridge current	200 mA	200 mA
e) Carrier gas pressure, bars	3.5 approx	1.5 approx
f) Flow rate,	40 to 70 ml/min	40 to 70 ml/min
g) Sample size	20	20

A-14.3.2 Sample Injection — Rinse the 50 μ l syringe with acetone twice and dry for two minutes or longer. Rinse the syringe twice before filling it with 20 microlitre of the synthetic standard. Inject the standard solution into the gas chromatograph and obtain a standard chart. Rinse the syringe twice with the material under test and inject 20 μ l into the chromatograph. It is imperative that the same injection technique is used and the syringe used in the operation is not used with any other type of solvent. It is very necessary to prevent contamination.

A-14.3.3 The elution order of the components is as follows:

- a) Methyl chloride
- b) Methylene chloride
- c) Chloroform

A-14.3.4 Calculation — Using a rule marked in fifty 0.02 units to 25 mm determine the peak height of each component. Refer to the calibration curves for the concentration of components present.

APPENDIX B

(Clause 5.1)

SAMPLING OF METHYLENE CHLORIDE (DICHLOROMETHANE), PURE AND TECHNICAL

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.1 Samples shall be taken in a protected place not exposed to damp air, dust or soot.

B-1.2 The sampling instrument shall be clean and dry.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.5 The samples shall be placed in suitable, clean, dry and air-tight glass bottles or other suitable containers on which the material has no action.

B-1.6 The sample containers shall be of such a size that they are almost three-fourths filled by the sample.

B-1.7 Each sample container shall be sealed air-tight after filling, and marked with full details of sampling, the date of sampling and details given under 3.2.

B-2. SCALE OF SAMPLING

B-2.1 Lot — All the containers in a single consignment of the material of the same grade drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared to consist of different batches of manufacture, the batches shall be marked separately and the groups of containers in each batch shall constitute separate lots.

B-2.2 For ascertaining the conformity of the material in any lot to the requirements of this specification, samples shall be tested for each lot separately.

B-2.3 The number of containers to be selected from the lot shall depend on the size of the lot and shall be in accordance with Table 2.

**TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED FROM
LOTS OF DIFFERENT SIZES**

(Clause B-2.3)

Lot Size N (1)	Sample Size n (2)
3 to 15	3
16 to 40	4
41 to 110	5
111 to 180	6
181 to 300	7
301 to 500	8
501 to 800	9
801 and above	10

B-2.3.1 These containers shall be selected at random. In order to ensure the randomness of selection, random number tables may be used (see IS : 4905-1968*). In case the random number tables are not available, the following procedure may be used:

Arrange all the containers in the lot in a systematic manner and starting from any one, count them as 1, 2, up to r where r is the integral part of N/n ; N and n being the lot size and sample size, respectively. Every r th container thus counted shall be withdrawn to constitute the sample.

B-3. INDIVIDUAL SAMPLES AND COMPOSITE SAMPLE

B-3.1 From each of the containers selected according to B-2.3, a representative portion of the material, about 300 ml in volume, shall be drawn. From each of these individual portions, an equal quantity of the material shall be taken and thoroughly mixed to constitute a composite sample not less than 1 000 ml in volume. The composite sample shall be transferred to clean bottles and labelled with full identification particulars of the sample.

B-4. NUMBER OF TESTS

B-4.1 Tests for the determination of the characteristics given in Table 1 shall be carried out on the composite sample.

B-5. CRITERIA OF CONFORMITY

B-5.1 For declaring the conformity of the lot to the requirements of all the characteristics, the test results on the composite sample shall meet the corresponding requirement specified.

*Methods for random sampling.

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

Quantity	Unit	Symbol
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

Quantity	Unit	Symbol
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

Quantity	Unit	Symbol	Definition
Force	newton	N	1 N = 1 kg. m/s ²
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m ²
Frequency	hertz	Hz	1 Hz = 1 c/s (s ⁻¹)
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m ²

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